PB-231 568

PROGRESS IN INSTRUMENTATION AND TECHNIQUES FOR MEASUREMENT OF AIR POLLUTANTS

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Office of Research and Development
Research Triangle Park, North Carolina

January 1974

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TECHNICAL REPORT DATA
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1. REPORT NO. EPA-650/2-74-015	2. PB 231 568
4. TITLE AND SUBTITLE Progress in Instrumentation and Techniques For Measurement of Air ~~Pollution~~ Pollutants	5. REPORT DATE January 1974 6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) Dr. A. H. Ellison	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Office of Research and Development U.S. Environmental Protection Agency Research Triangle Park, N. C. 27711	10. PROGRAM ELEMENT NO. 1AA010 11. CONTRACT/GRANT NO.
12. SPONSORING AGENCY NAME AND ADDRESS Office of Research and Development U.S. Environmental Protection Agency Washington, D. C. 20460	13. TYPE OF REPORT AND PERIOD COVERED In-house 14. SPONSORING AGENCY CODE
15. SUPPLEMENTARY NOTES	

16. ABSTRACT

This is a brief report on instrumentation and techniques for the measurement of air pollutants. It covers the techniques and instrumentation that have been promulgated by EPA along with air pollution standards, those that are in use by EPA for developing standards, and those that are being developed by EPA to meet current or future needs. Descriptions of these techniques are brief but reference to the detailed procedures are given.

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17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Air Pollution	Hydrocarbons		
Air Pollution Measurement	Carbon monoxide		
Air Pollution Measurement	Hydrogen Sulfide		
Calibration	Mercury		
Particulate Matter	Beryllium		
Sulfur Oxides	Asbestos		
Nitrogen Oxides			
Photochemical Oxidants			

18. DISTRIBUTION STATEMENT Release unlimited	19. SECURITY CLASS *(This Report)* Unclassified 20. SECURITY CLASS *(This page)* Unclassified

EPA Form 2220-1 (9-73)

36. Development of a Method for the Determination of Asbestos in Ambient Air. Prepared for EPA by Battelle Memorial Institute under Contract number CPA 22-69-110. August 1971.

37. National Bureau of Standards. Washington, D.C. Technical Note 585. January 1972. p. 6-10.

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EPA-650/2-74-015

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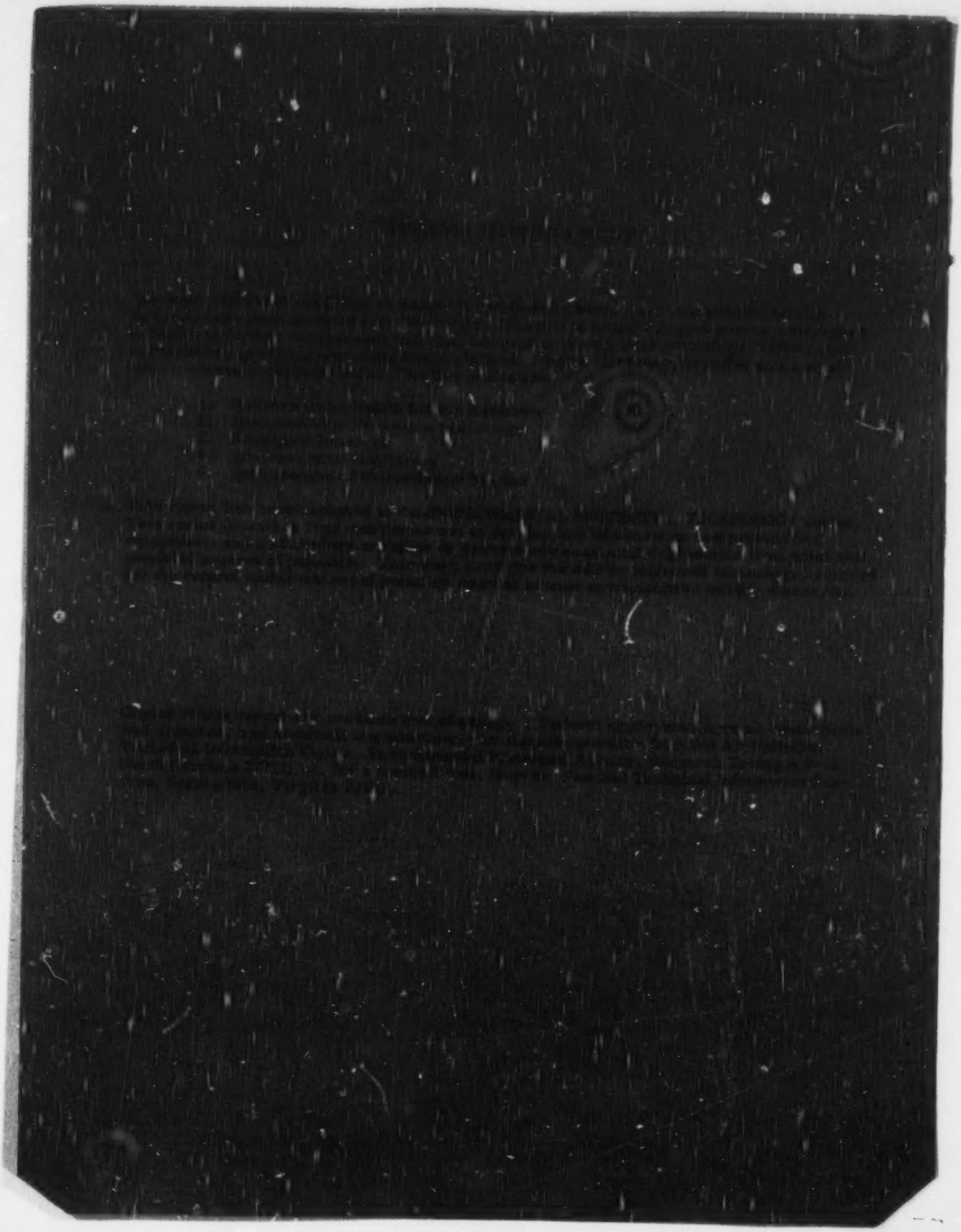
This report has been reviewed by the Office of Research and Development, Environmental Protection Agency, and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Environmental Protection Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.



PREFACE

This discussion has been limited to EPA's air pollution measurement technique and instrumentation research and development program; however, this is not to imply that research conducted in other government agencies and in the private sector have not contributed to the EPA program. In fact, much of EPA's program has involved the evaluation and application of technology developed elsewhere. The bibliography given contains only the key references. These references in turn contain many additional references.

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PROGRESS IN INSTRUMENTATION AND TECHNIQUES FOR MEASUREMENT OF AIR POLLUTANTS

INTRODUCTION

The Clean Air Amendments of 1970, which became law in December of that year, directed the Environmental Protection Agency (EPA) to bring air pollution under control through the promulgation of numerous national air pollution standards. It authorized the promulgation of ambient air quality standards, standards for new stationary sources of air pollution, standards for hazardous air pollutants, standards for mobile source emissions, and standards for fuels and fuel additives. In anticipation of the 1970 legislation, EPA's predecessor in the air pollution control program, the National Air Pollution Control Administration (NAPCA), which was part of the Department of Health, Education, and Welfare, started work on a plan for the research and development needed to provide appropriate measurement techniques and instrumentation for developing and supporting the various standards to be promulgated. The plan was prepared under contract with Esso Research and Engineering Company and was completed with EPA funding and published in December of 1971.[1]

It had been established by NAPCA that the plan should include research for and development of measurement techniques for the air pollutants listed below:

1. Oxides of sulfur (SO_2, SO_3, H_2SO_3)
2. Oxides of nitrogen (NO_x, NO, NO_2, HNO_3)
3. Particulate matter (size distribution, chemical composition)
4. Asbestos
5. Mercury
6. Beryllium
7. Carbon monoxide
8. Nonmethane hydrocarbons
9. Certain specific hydrocarbons
10. Polychorinated biphenyls
11. Polynuclear organic matter
12. Reactive hydrocarbons
13. Hydrogen chloride
14. Manganese
15. Selenium
16. Arsenic
17. Phosphoric acid
18. Chlorine
19. Hydrogen fluoride
20. Hydrogen sulfide

21. Mercaptans
22. Ammonia and amines
23. Organic acids
24. Aldehydes
25. Odor
26. Photochemical oxidants
27. Copper
28. Zinc
29. Boron
30. Tin
31. Lithium
32. Chromium
33. Vanadium
34. Cadmium
35. Lead
36. Aeroallergens

Two determinations were then made for each of those pollutants: the type of air pollution standard that probably would be needed and the type of measurement methodology that would be required to develop and support an appropriate standard. Of course, the need for appropriate sampling procedures and supporting measurements such as those required to calculate mass rates from measured concentrations of a pollutant in a source emission had to be addressed in the plan.

For the most part, the air pollution measurement technique and instrumentation research and development program of EPA is being carried out according to plan. Because of funding limitations, however, the various tasks will be completed later than indicated in the original plan. Also, research and development requirements not envisioned at the time the plan was prepared are being added to the program as needed.

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PROMULGATED AND PROPOSED STANDARDS

As of the summer of 1973, National Ambient Air Quality Standards had been promulgated for SO_2, particulate matter, photochemical oxidants, hydrocarbons, nitrogen dioxide, and carbon monoxide.[2] Standards of performance for new stationary sources had been promulgated for particulate matter, sulfur dioxide, and nitrogen oxides from fossil-fuel-fired steam generators; particulate matter from large incinerators; particulate matter from Portland cement plants; nitrogen oxides from nitric acid plants; and sulfur dioxide and acid mist from sulfuric acid plants.[3] New-source performance standards had been proposed for particulate matter from asphalt concrete plants; particulate matter, carbon monoxide, sulfur dioxide, and hydrogen sulfide from petroleum refineries; hydrocarbons from storage vessels for petroleum liquids; particulate matter from secondary lead smelters; particulate matter from secondary brass and bronze ingot production plants; and particulate matter from sewage treatment plants.[4]

By this same time, hazardous air pollutant standards had been promulgated for asbestos from asbestos mines, asbestos mills, manufacturing operations, the repair or demolition of buildings or structures, spraying operations, and roadway surfacing operations; for beryllium from machine shops, ceramic plants, propellant plants, foundries, extraction plants, and incinerators for disposal of toxic substances; and mercury from ore processing plants and chloralkali plants.[5]

Also by the summer of 1973, emission standards for carbon monoxide, hydrocarbons, and nitrogen oxides from light-, medium-, and heavy-duty vehicles had been promulgated. A standard for smoke from new heavy-duty diesel engines had been promulgated.[6] Regulations had also been promulgated,[7] and others proposed,[8] for lead and phosphorus additives in gasoline but measurement techniques for these additives had not yet been specified. Emission standards had been promulgated for certain types of aircraft engines[9] and proposed for other types.[10]

Although not proposed or promulgated to date, appropriate standards are being considered for other pollutants among the 36 listed above. Detailed measurement procedures are usually included as part of the standards, but some standards rely on the estimated effectiveness of required control measures-e.g., floating roofs for storage vessels for petroleum liquids-rather than on monitoring of emissions by means of specified techniques.

Air pollution control legislation prior to 1970 provided for the achievement of adequate air quality by state and local agencies on the basis of Federal criteria.[11-16] It also contained authority for establishing emission standards for light-duty vehicles. Thus the research program for development of measurement techniques and instrumentation in support of pre-1970 legislation was concerned mostly with air quality and light-duty

vehicle emissions. This program has borne fruit and it will be apparent in the discussion below that great progress has already been made in these areas.[17,18] The 1970 legislation called for the control of emissions from stationary sources as rapidly as possible and authorized EPA to set standards based on the best control technology available. A major effort had to be mounted to provide stationary source emission measurement techniques in support of the additional control activities called for by the 1970 legislation.[19]

MEASUREMENT STRATEGIES

At present, various measurement strategies are being considered for use in the air pollution control program. For example, air pollutant levels in an urban area or a small locality may be measured by means of one or several monitoring stations or, if current research is successful, it may be possible to measure pollutant levels through the use of long-path spectroscopic techniques. Stack emissions from stationary sources may be measured by extraction of a sample of the effluent gas followed by conventional analysis, or by the use of an across-the-stack optical technique, or by remote measurement of the stack plume. Not only do control activities require pollutant measurements, but studies on the effects of air pollutants on human subjects in an urban area require them as well. Data on health-related pollution may be obtained through multistation monitoring of pollutant levels in the locality where the subjects live and work or by providing the individuals with miniaturized portable instruments that can record and integrate their exposure to pollution. There are needs for the continuous measurement of air pollutant levels in the air or in a source emission that would require appropriate instrumentation, and there are needs that require the periodic collection of samples (in a bubbler, on a solid absorbent, on a filter, in an evacuated cyclinder, or in an expandable plastic bag), followed by analysis in a central laboratory.

PARTICULATE MATTER

Particulate matter in ambient air is measured by means of the high-volume sampler.[2] Results of relatively high precision are obtainable by this method, as a collaborative test has shown.[20] It is generally agreed, however, that improved measurement methodology is needed for ambient air particulate matter. Methods that provide particle size fractionation, at least into two size fractions, are the objects of much of the research.

Particulate matter in emissions from stationary sources is measured gravimetrically after collection with the EPA sampling train.[3] It is known that under certain conditions particulate matter can form in this sampling system. Particulate matter can also remain within the sampling probe; thus a procedure for removing and weighing this portion of the collected sample is needed. There is much interest in the development of methodology for the continuous measurement of particulate matter in ambient air and source emissions; one program is aimed at developing suitable instrumentation based on the attenuation of beta radiation by particulate matter collected on a filter.[21]

The chemical characterization of particulate matter is important not only as a means of identifying sources of atmospheric particulate matter but also for identifying specific chemical elements or compounds for which standards might be promulgated. Of the several techniques for chemical analysis of particulate matter that are possible, x-ray fluorescence appears especially promising.[22] This technique may supplement or replace previously used techniques such as atomic absorption spectrophotometry, emission spectroscopy, and neutron activation analysis.

Numerous problems have been encountered in the use of filters for collecting particulate matter. No one filter material is suited to all tasks. Chemical analysis of particulate matter by X-ray fluorescence requires very low background levels of impurities. Mass measurement by beta attenuation requires low mass per unit area for maximum sensitivity; however, the strength necessary for use and handling must be maintained. Adsorption or absorption by filters of materials in the gas phase (i.e., water and organics) has been a major unresolved problem. Several active programs are addressed at solving these problems.

SULFUR OXIDES

Sulfur dioxide in ambient air is measured by the pararosaniline method.[2] A collaborative test of this method has shown that satisfactory precision is obtained.[23] Sulfur dioxide (SO_2) in ambient air can be measured continuously by means of a flame photometric detection system. The detector measures total sulfur and therefore can be used only when it is known that SO_2 is the major (<95%) sulfur-containing species present[24] or after sulfur compounds are separated by gas-liquid chromatography.[25] SO_2 in fossil-fuel-fired power plants is measured by collection of SO_2 in 3% hydrogen peroxide and subsequent titration using barium perchlorate with a thorin indicator.[3] A sampling train is used in which SO^3 and H_2SO^4 are removed by collection in 80% isopropanol. For the measurement of SO_2, SO_3, and H_2SO_4 mist from sulfuric acid plants, a filter is added between the 80% isopropanol collection solution and the 3% hydrogen peroxide collection solutions. Sample probe washings, 80% isopropanol collection, and filter washings are considered to be sulfuric acid mist; the fraction collected in 3% hydrogen peroxide is SO_2. Both fractions are analyzed for sulfur oxide content by the barium perchlorate titration using thorin as an indicator.[3] Sulfur dioxide in power plant stack emissions may be measured continuously by nondispersive infrared spectroscopy (NDIR) with suitable sample conditioning to remove particulate matter and water vapor and to reduce gas temperature to near ambient.[26]

NITROGEN OXIDES

Nitrogen dioxide (NO_2) in ambient air has been measured by a 24-hour method that involves collection in a sodium hydroxide solution to form a stable solution of sodium nitrite that is then determined colorimetrically by a diazotization reaction; however, this method has been found to be inadequate because of variable collection efficiency and the interference of NO.[27] Intensive work is being done to develop and evaluate improved collection techniques.[28] These efforts involve the evaluation of several collection solutions such as those described by Christie,[29] Saltzman,[30] and Mulik et al.[31] To be adequate, a method must have a high degree of specificity and a constant collection efficiency. Continuous measurement of NO_2 in ambient air is accomplished by a procedure based on the chemiluminescent reaction between NO and ozone.[28] NO_2 is converted to NO in a suitable sampling system. The sample is alternately passed directly to the chemiluminescence detection chamber and indirectly to the detection chamber via the converter so that the technique measures first NO and then NO plus NO_2, permitting, by difference, a determination of NO_2. The nitrogen oxides stationary source and mobile source emission standards are for NO_x, that is, NO_2 plus NO. Mobile source emissions of NO_x are measured by the chemiluminescence procedure described for ambient air.[32] The sampling system uses a thermal reactor for the conversion of NO to NO_2. Nitrogen oxides in power plant emissions are collected in a dilute sulfuric acid-hydrogen peroxide solution and measured colorimetrically using phenol disulfonic acid.[3] An electrochemical technique and nondispersive ultraviolet and NDIR spectroscopy have been proposed as methods for the continuous measurement of NO_x in power plant emissions.[33]

PHOTOCHEMICAL OXIDANTS

It has been shown that after correction for NO_2 ozone comprises virtually all of photochemical oxidant. Consequently, the ambient air standard for photochemical oxidants is based on a measurement technique for ozone. The measurement technique uses the chemiluminescent reaction that occurs between ozone and ethylene. This technique permits the continuous measurement of ozone,[2] as well as the 1-hour measurement on which the air quality standard for ozone is based. A collaborative test of this chemiluminescence method showed that satisfactory precision can be obtained.[34] Since oxidant is formed in the atmosphere by photochemical reactions involving hydrocarbons and nitrogen oxides, there is no need for source measurements of ozone. Rather, photochemical oxidants are controlled by limiting the emission of hydrocarbons.

HYDROCARBONS

Hydrocarbons in the ambient air are measured by a flame ionization detector.[2] Since methane does not participate in photochemical reactions that form oxidants, values for methane are subtracted from the total hydrocarbon measurement. This is done by means of a stripper column and an analytical column that remove interfering substances and allow only methane to pass through. Thus, the sample is alternately passed directly to the detector and indirectly to the detector through the stripper and analytical columns, which retain all hydrocarbons except methane.

Hydrocarbons in mobile source emissions are measured by a flame ionization detector or by NDIR.[6] Flame ionization detection is used for aircraft and all vehicles; however, NDIR can be used for heavy-duty vehicles. The measurement is for total hydrocarbons. Methane has been a minor constituent of mobile source hydrocarbon emissions, so that no correction for methane is presently made. However, the catalytic devices that will be used in future vehicles tend to result in less effective oxidation of methane. Methane may then constitute a larger fraction of the hydrocarbons emitted from vehicles. Further, it is now believed that other hydrocarbons such as ethane, propane, acetylene, and benzene may be relatively unreactive in the formation of photochemical oxidant. Thus, work to develop measurement techniques for these hydrocarbons is under way should it be deemed advisable to subtract them along with methane from total hydrocarbon measurements. No stationary source emission standards for hydrocarbons have been promulgated or proposed by EPA that require hydrocarbon measurement.

CARBON MONOXIDE

Carbon monoxide in ambient air[2] and in mobile[6] and stationary[4] source emissions is measured by nondispersive infrared spectroscopy. A collaborative test of the method for ambient air analysis showed satisfactory precision of the method.[35]

HYDROGEN SULFIDE

Hydrogen sulfide emissions from petroleum refineries are measured by collection in alkaline cadmium hydroxide solution to form cadmium sulfide. The precipitated cadmium sulfide is then dissolved in a known volume of HCl-acidified iodine solution. The iodine consumed is a measure of the H_2S present.[4]

HAZARDOUS AIR POLLUTANTS: MERCURY, BERYLLIUM, AND ASBESTOS

Mercury, beryllium, and asbestos have been designated hazardous air pollutants and, as stated above, emission standards for them have been promulgated.

Stationary source mercury emissions, both particulate and gaseous, are measured after appropriate sampling by collection in an iodine monochloride solution. The mercury collected is reduced to elemental mercury in basic solution by dihydroxylamine sulfate. Mercury is purged from the solution using a zero-grade air stream and analyzed using an atomic absorption spectrophotometer in the flameless mode.[5]

Beryllium is measured after appropriate sampling by digestion in an acid solution and subsequent analysis by means of atomic absorption spectrophotometry.[5]

An appropriate method for measuring asbestos emissions is not available. An electron microscope technique is used to determine asbestos in filtered ambient air samples[36] but this technique is not rapid enough to support an emission standard. Current work is under way to provide an adequate technique for measuring source emissions of asbestos.

INSTRUMENT AND METHOD CALIBRATION

Calibration of air pollution measurement techniques is exceedingly important and in many cases requires close attention to experimental details. Various types of air pollutant reference samples are in use. Permeation tubes for SO_2 are available as standard reference materials from the National Bureau of Standards;[37] a similar device for NO_2 is being perfected.[38] Calibrated ozone generators are being used.[39] Small gas cylinders containing known amounts of carbon monoxide[40] or hydrocarbons[41] are being evaluated as reference standards. A large batch of atmospheric particulate matter is being collected so that it can be completely characterized physically and chemically and then used as a reference for particulate matter measurement techniques.[42] A gas-phase titration of NO and ozone to produce NO_2 has been developed so that the calibration of techniques for NO, NO_2, and ozone can be accomplished with a reference sample of any one of the three constituents.[28]

RESEARCH TECHNIQUES

In addition to the measurement techniques that have been developed for the purpose of determining compliance with an air pollution standard, measurement techniques are needed for the research program that must be conducted to develop the information needed in the setting of a standard. Virtually the entire spectrum of analytical techniques is used, including spectroscopy-particularly infrared spectroscopy[43]-gas chromatography,[44] electron microscopy, x-ray fluorescence, x-ray diffraction, mass spectroscopy, emission spectroscopy, and neutron activation analysis. For the most part, these methods employ laboratory instruments, so that techniques must be used that will permit samples to be collected in the field but brought to the laboratory for analysis. In some instances, however, it is possible to install equipment of this type in mobile laboratories so that measurements can be made in the field.

Remote and long-path optical techniques for measurement of pollutants in ambient air or in stationary source effluents are being developed.[45] In some instances, the instruments utilized in these techniques use broad-band artificial light sources; in other cases, the sun's radiation is used; and, in others, laser sources are used. Research and development costs for this type of instrumentation are high, and significant technical problems exist in certain applications. However, the potential effectiveness of air pollution measurement and monitoring by means of these techniques justifies a reasonable effort to investigate their usefulness.

The resolution of the problem of preventing environmental degradation in nonurban areas[46] will require more sensitive measurement techniques than presently are available. Also, it will be necessary to use these techniques to establish a base of data against which degradation can be assessed.

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